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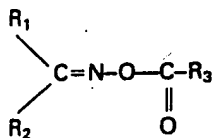
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(71) Applicant Daicel Chemical Industries Ltd., (Incorporated in Japan), 1-Banchi Teppō-cho, Sakai-shi, Osaka, Japan	(56) Documents cited None
(72) Inventors Masanori Itoh, Fumio Takenaka, Kouzi Tohya	(58) Field of search C3P
(74) Agent and/or Address for Service W. P. Thompson & Co., Coopers Building, Church Street, Liverpool L1 3AB	

(54) Photopolymerization Initiator

(57) The initiator composition is effectively used for a photopolymerizable resin composition which comprises a photopolymerizable compound having a double bond for polymerization and a polymer binder and comprises (A) thioxanthone or a substituted thioxanthone and (B) an oxime ester of the formula:



(III)

wherein R_1 and R_2 may be the same or different and each represent an alkyl group having 1 to 10 carbon atoms, a substituted or unsubstituted phenyl, naphthyl, anthryl, pyridyl or quinolyl group or R_1 and R_2 may be bonded together to form a ring, and R_3 represents an alkyl group having 1 to 5 carbon atoms or a substituted or unsubstituted aryl group.

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SPECIFICATION

Photopolymerization initiator

5 The present invention relates to a photopolymerization initiator. More particularly, the invention relates to a photopolymerization initiator to be used for a photopolymerizable resin composition which is cured by photopolymerization in those parts exposed to light activation and which has an excellent adhesion to a base material.

Photopolymerizable resin compositions have been used as starting materials for inks cured by light activation, paints and photoresists. They have a serious defect that the curability of coating films of them is poor upon exposure to light. The thickness of the coating film exerts a great influence on the curability. For example, the coating film of photoresist or screen printing ink may be as thick as 10 to 100 μm and, therefore, the inner surface of the coating film is not cured sufficiently. This phenomenon is particularly in evidence when a pigment or a dye is incorporated in the photopolymerizable resin composition and the coating film surface remains tacky. Thus, the expected curability is not obtained.

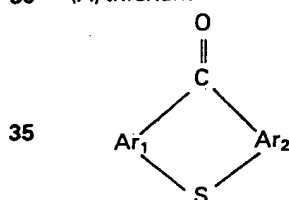
Processes have been proposed for solving the problem of poor curability by using a combination of photopolymerization initiators such as a combination of benzophenone or its derivative with 4,4'-bis-(diethylamino)benzophenone (Japanese Patent Publications Nos. 37902/1978 and 25943/1979) and a combination of xanthone with a 4,4'-bis(dialkylamino)-benzophenone (Japanese Patent Laid-Open No. 92246/20 1977).

Though these combined photopolymerization initiators are effective, their effects are still insufficient particularly when a dye or a pigment is used, since with a coating film thickness of more than 10 μm , the curability is insufficient and the adhesion to a base material is reduced.

The inventors have found that a photopolymerizable composition have an excellent coating film curability and excellent adhesion to a base material can be obtained when a mixture of thioxanthone, or its derivative, with an oxime ester is used as the photopolymerization initiator. The present invention has been completed on the basis of this finding.

According to the present invention there is provided a composition for photopolymerization initiation which comprises:

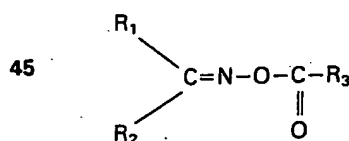
30 (A) thioxanthone or a derivative thereof of the general formula:



(I)

in which Ar_1 and Ar_2 each represent an aromatic ring or a substituted aromatic ring, and

(B) an oxime ester of the general formula:



(II)

50 wherein R_1 and R_2 may be the same or different and each represents an alkyl group having 1 to 10 carbon atoms, a substituted or unsubstituted phenyl, naphthyl, anthryl, pyridyl or quinolyl group or R_1 and R_2 may be bonded together to form a ring, and R_3 represents an alkyl group having 1 to 5 carbon atoms, or a substituted or unsubstituted aryl group.

The photopolymerization initiator of the present invention may be used in a photopolymerizable resin composition together with a photopolymerizable compound, a polymer binder and other additives.

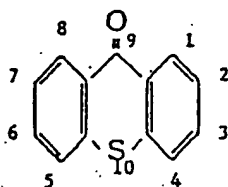
According to the present invention there is further provided a photopolymerizable resin composition which comprises 5 to 80 percent by weight of one or more photopolymerizable compounds having a double bond for polymerization, 0.01 to 20 percent by weight of the photopolymerizable initiator composition of the present invention, 0 to 94.99 percent of a polymer binder, and the balance, if any, comprising other photopolymerizable resin composition additives.

The photopolymerization initiator of the present invention is suitable for use with a photopolymerizable compound having at least one polymerizable double bond in the molecule. Typical examples of these compounds include styrene compounds such as styrene *per se*, α -methylstyrene and chlorostyrene; mono-functional (meth)acrylates such as methyl (meth)-acrylate, ethyl (meth) acrylate, n- and i- propyl (meth)-acrylates, n-, sec- and t-butyl (meth)-acrylates, tetrahydrofurfuryl (meth)acrylate, 2-ethylhexyl (meth)acrylate,

2-ethylhexyl (meth)-acrylate, lauryl (meth)acrylate, stearyl (meth)-acrylate, butoxyethyl (meth)acrylate, cyclohexyl (meth)acrylate, phenoxyethyl (meth)acrylate, methoxyethyl (meth)acrylate, ethoxyethyl (meth)acrylate, polyethylene glycol (meth)acrylate, and polypropylene glycol (meth)acrylate; bifunctional (meth)acrylates such as ethylene glycol di(meth)acrylate, diethylene glycol di(meth)acrylate, triethylene glycol di(meth)acrylate, propylene glycol di(meth)acrylate, dipropylene glycol di(meth)acrylate, polyethylene glycol di(meth)acrylate, polypropylene glycol di(meth)acrylate, 1,4-butanediol di(meth)acrylate, neopentyl glycol di(meth)acrylate and 1,6-hexanediol di(meth)acrylate; and trifunctional or higher functional (meth)acrylates such as trimethylolpropane tri(meth)acrylate, trimethylolethane tri(meth)acrylate and pentaerythritol tetra(meth)acrylate. These polymerizable compounds may be used either alone or in combination. They are used in an amount of 5 to 80 wt.%, preferably 10 to 50 wt.%, based on the weight of the photopolymerizable resin composition.

The polymer binders include polystyrene, polymethyl methacrylate, methyl methacrylate/ethyl acrylate copolymer, poly(meth)acrylic acid, styrene/(meth)acrylic and copolymer, (meth)acrylic acid/methyl methacrylate copolymer, polyvinyl butyral, cellulose esters, polyacrylamide and saturated polyesters. The binder is selected depending on the purpose.

The invention provides use of two compounds in combination as an initiator for photopolymerization. In one embodiment an initiator composition of the invention comprises thioxanthone having the formula I above, or a derivative thereof, having a substituent on at least one of aromatic rings, and an oxime ester having a specific formula (II).



The aromatic rings Ar₁ and Ar₂ in the formula I above may be substituted with one or more of the following substituents: hydrogen; halogens such as chlorine or bromine; alkyl groups having 1 to 12 carbon atoms which may be substituted with a hydroxy or chlorine, such as methyl, ethyl, n-propyl, isopropyl, n-butyl, sec-butyl, t-butyl, n-amyl, isoamyl, n-hexyl, 1,1-dimethylbutyl, 1-ethyl-2-methylpropyl, 2-ethylbutyl, 1,3-dimethylbutyl, 4-methylpentyl, n-heptyl, 1-methylhexyl, 1-ethyl-1,2-dimethylpropyl, n-octyl, 2-ethylhexyl, 2,2,4-trimethylpentyl, n-nonyl, 1,3,5-trimethylhexyl, n-dodecyl, chloromethyl, 2-chloroethyl and 2-hydroxyethyl groups; alkoxy groups having 1 to 8 carbon atoms which may be substituted with a hydroxy, chlorine alkoxy group having 1 to 4 carbon or alkoxy-alkoxy group in which each alkoxy group has 1 to 4 carbon atoms, such as methoxy, ethoxy, n-propoxy, isopropoxy, sec-butoxy, n-butoxy, n-pentyloxy, isopentyloxy, 2-ethylhexyloxy, allyloxy, 2-chloroethoxy, 2-hydroxypropoxy, glycidyloxy, 2-hydroxyethoxy, methoxyethoxy, butoxyethoxy, ethoxyethoxyethoxy and benzyloxy groups; acyloxy groups having 1 to 18 carbon atoms, such as acetyloxy, propionyloxy, acryloyloxy, methacryloyloxy, butyroyloxy, isobutyroyloxy, nonanoyloxy, crotonoyloxy, decanoyloxy, dodecanoyloxy, stearyloxy, fumaroyloxy, phthaloyloxy, cinnamoyloxy and benzyloxy; alkoxycarbonyl groups having 1 to 8 carbon atoms which may be substituted with a hydroxy or chlorine, such as methoxycarbonyl, ethoxycarbonyl, 2-hydroxypropoxycarbonyl, 2-chloroethoxycarbonyl, n-butoxycarbonyl, 2-ethylhexylcarbonyl and octyloxycarbonyl; in addition cyclohexyl, phenyl, phenoxy, vinyloxy, allyloxy, glycidyloxy and benzyloxy groups may be used. The aromatic rings Ar₁ and Ar₂ may be substituted with the same substituents or each of Ar₁ and Ar₂ may be substituted with different substituents. The manner of substitution does not limit the present invention.

The oxime esters of the present invention are shown by the above general formula (II) in which R₁ and R₂ may be the same or different and they are as defined in the following groups 1) to 4):

- 1) a straight-chain or branched alkyl group having 1 to 10, preferably 1 to 5 carbon atoms,
 - 2) a phenyl, naphthyl or anthryl group which may be substituted with a straight-chain or branched alkyl group having 1 to 10, preferably 1 to 5, carbon atoms, an alkoxy group having 1 to 5 carbon atoms, a halogen, an amino, dimethylamino, diethylamino, or mercapto group,
 - 3) a pyridyl group, for example 2-pyridyl group, a quinolyl group, for example 6-quinolyl group or pyridyl or quinolyl group substituted with the substituents set forth as in 2) above, and
 - 4) a ring formed by bonding R₁ and R₂ together, such as; for example, cyclohexanone, anthrone, 10-methyl-9(10H)-acridone, piperidone, or fluorenyl groups, e.g. 9-fluorenone, which group may be substituted with the substituents set forth as in 2) above, and R₃ is as defined in the following groups 5) and 6):
 - 5) an alkyl group having 1 to 5 carbon atoms, for example methyl, ethyl, propyl, i-propyl, butyl or t-butyl group, and
 - 6) an aryl group, for example, phenyl groups, benzoyl, toluoyl, chlorobenzoyl, nitrobenzoyl, methoxybenzoyl, naphthyl and anthroquinone, which may be unsubstituted or substituted with the substituents set forth in 2) above.
- The above-mentioned oxime esters include various ketone-O-acyloximes. Examples of them include

dimethyl ketone-O-acetyloxime, diethyl ketone-O-acetyloxime, acetophenone-O-acetyloxime, acetophenone-O-benzoyloxime, acetophenone-O-(p-methylbenzoyl)oxime, p-methylacetophenone-O-(p-tolylcarbonyl)oxime, 2-naphthyl methyl ketone-O-acetyloxime, 2-naphthyl phenyl ketone-O-propylcarbonyloxime, 9-anthryl methyl ketone-O-acetyloxime, 2-naphthyl methyl ketone-O-benzoyloxime, 5 benzophenone-O-acetyloxime, benzophenone-O-phenylacetyloxime, benzophenone-O-(p-chlorobenzoyl)oxime, p-dimethylaminoacetophenone-O-acetyloxime, p,p'-bis-(dimethylamino)benzophenone-O-acetyloxime, p,p-bis(diethylamino—benzophenone-O-benzoyloxime, fluorenone-O-(p-methylbenzoyl)oxime, fluorenone-O-acetyloxime, 2-pyridyl methyl ketone-O-acetyloxime, di(2-pyridyl) ketone-O-acetyloxime, 6-quinolyl methyl ketone-O-ethylcarbonyloxime and 6-quinolyl phenyl 10 ketone-O-acetyloxime. These oxime esters by no means limit the present invention.

These oxime esters, as with the above-mentioned thioxanthone and its derivatives, may be used either alone or in the form of a mixture of two or more of them.

The photopolymerization initiator of the present invention may be used in combination with a known photosensitizer such as benzoin, benzoin alkyl ether, benzophenone, benzyl-2,4,5-triphenylbiimidazole or a 15 derivative thereof.

The ratio of thioxanthone or its derivative to the oxime ester used in the present invention is selected depending on the varieties of them and/or the photopolymerizable compound. Preferably, the weight ratio of thioxanthone or its derivative to the oxime ester is in the range of 1/100 to 10/1.

The amount of the photopolymerization initiator of the present invention is in the range of 0.01 to 20 wt.%, preferably 1 to 10 wt.%, based on the photopolymerizable resin composition (solid). 20

A preferred embodiment of the photopolymerizable resin composition comprises 30 to 100 weight percent in total of the photopolymerizable compound and the initiator composition, and zero to 70 weight percent of the polymer binder.

The photopolymerizable resin composition containing the photopolymerization initiator of the present 25 invention is applied to a base material by a known method and then exposed to an activating ray to induce the photopolymerization reaction.

The sources of the light used include far ultraviolet ray sources, e.g. Hg-He or Hg-Xe lamps, low-pressure mercury lamps, or excimer lasers, (a Tradename), each having an intensive emission at 200 to 300nm. Sources having an emission spectrum of 300 nm or higher, e.g. Hg lamps, xenon lamps or high-pressure mercury 30 lamps may be used. N₂ lasers, argon lasers, and He-Ne lasers may also be used.

Specific embodiments of the present invention will now be described with reference to the following Examples.

Example 1

35 A photopolymerizable resin composition was obtained by mixing the following components:

trimethylolpropane triacrylate	5 g	
2-chlorothioxanthone	0.01 g	
4-methylbenzophenone oxime acetate	0.25 g	
40 methyl ethyl ketone	5 g	40

5 ml of the composition was placed in a Pyrex glass test tube and exposed to the light of a 2 kW ultra-high pressure mercury lamp from a distance of 50 cm for 1 min. The polymer adhered, in the form of a film, to the inner wall of the exposed part of the tube. When the polymer was taken out and placed in acetone, it was not 45 dissolved therein.

Example 2

A photopolymerizable resin composition was obtained by mixing the following components:

50 poly(methyl methacrylate)	50 g	50
trimethylolpropane triacrylate	30 g	
2-chlorothioxanthone	0.05 g	
4-methylbenzophenone oxime acetate	1.5 g	
55 methyl ethyl ketone	150 g	55

The composition was applied uniformly to a polyethylene glycol terephthalate film base having a thickness of 10 μ m by means of an applicator and then dried at 75°C to form a light-sensitive layer having a thickness of 25 μ m. This product was exposed to a light of a 2 kW ultra-high pressure mercury lamp at a distance of 50 cm for 30 sec. When the polyethylene glycol terephthalate film was bent at a sharp angle, cracks were formed in 60 the photosensitive layer to indicate that it had been cured sufficiently.

Example 3

A photopolymerizable resin composition was obtained by mixing the following components:

	poly(methyl methacrylate)	50 g	
5	trimethylolpropane triacrylate	15 g	5
	tetraethylene glycol diacrylate	10 g	
	benzotriazole	0.2 g	
	N-methyldiethanolamine	0.05 g	
	Victoria Blue	0.04 g	
10	2-chlorothioxanthone	0.1 g	10
	4-methylbenzophenone oxime acetate	1.5 g	
	methyl ethyl ketone	120 g	

- The composition was applied uniformly to a polyethylene glycol terephthalate film base having a thickness of 25 μm and dried at 75°C to obtain a photosensitive film having a thickness of 25 μm . The photosensitive layer was applied to a copper plate, having the surface previously washed with acetone, by means of a rubber roll heated to 100°C, to form a laminate. Then, a Stouffer step tablet (Tradename of Stouffer Graphic Arts Instrument Corporation), was placed thereon and exposed to a light of an ultra-high pressure mercury lamp (2 kW) at a distance of 50 cm for 10 sec. The non-exposed part was washed away with 1,1,1-trichloroethane to effect the development. A high-quality image was obtained until the seventh step.

Example 4

A photopolymerizable resin composition was obtained by mixing the following components:

25	poly(methyl methacrylate/butyl acrylate/methacrylic acid) (61/17/22 wt.%)	50 g	25
	trimethylolpropane triacrylate	30 g	
	benzotriazole	0.2 g	
30	N-methyldiethanolamine	0.05 g	30
	Victoria Blue	0.04 g	
	2-chlorothioxanthone	0.1 g	
	4-methylbenzophenone oxime acetate	1.5 g	
	methyl ethyl ketone	45 g	
35	dioxane	60 g	35

- The composition was applied to a film base and dried in the same manner as in Example 3 to obtain a sensitive layer having a thickness of 25 μm . After the exposure in the same manner in Example 3, followed by development with a 1% aqueous sodium carbonate solution at 40°C, a high-quality image was obtained until the sixth step.

Example 5

A photopolymerizable resin composition was obtained by mixing the following components:

45	poly(methyl methacrylate)	50 g	45
	trimethylolpropane triacrylate	15 g	
	tetraethylene glycol diacrylate	10 g	
	2-phenylamino-4,6-dithiol-s-triazine	0.2 g	
	N-methyldiethanolamine	0.05 g	
50	Victoria Blue	0.04 g	50
	2-chlorothioxanthone	0.1 g	
	4-methylbenzophenone oxime acetate	1.5 g	
	o-toluenesulfonamide	2.4 g	
	p-toluenesulfonamide	3.6 g	
55	methylethyl ketone	120 g	55

A high-quality image was obtained until the fifth step in the same manner as in Example 3.

Example 6

A photopolymerizable resin composition was obtained by mixing the following components:

	poly(methyl methacrylate)	50 g	
5	trimethylolpropane triacrylate	16 g	5
	tetraethylene glycol diacrylate	13 g	
	2-phenylamino-4,6-dithiol-s-triazine	0.2 g	
	N-methyldiethanolamine	0.05 g	
	Victoria Blue	0.04 g	
10	2,2'-di(o-chlorophenyl)-4,4',5,5'-tetraphenylbisimidazole	1.5 g	10
	Leucocrystal Violet	0.15 g	
	2-chlorothioxanthone	0.1 g	
	4-methylbenzophenone oxime acetate	1.5 g	
15	o-toluenesulfonamide	2.4 g	15
	p-toluenesulfonamide	3.6 g	
	methylethylketone	120 g	

A high-quality image was obtained until the 8th step in the same manner as in Example 3.

20 20

Example 7

A photopolymerizable resin composition was obtained by mixing the following components:

	poly(methylmethacrylate)	50 g	
25	trimethylolpropane triacrylate	16 g	25
	tetraethylene glycol diacrylate	13 g	
	N-methyldiethanolamine	0.05 g	
	Victoria Blue	0.04 g	
	thioxanthone	0.5 g	
30	4-methylbenzophenone oxime acetate	1.5 g	30
	o-toluenesulfonamide	2.4 g	
	p-toluenesulfonamide	3.6 g	
	benzotriazole	0.2 g	
	methylethyl ketone	120 g	

35 35

A high-quality image was obtained until the 8th step in the same manner as in Example 3.

Example 8

A photopolymerizable resin composition was obtained by mixing the following components:

40	poly(methyl methacrylate)	50 g	40
	trimethylolpropane triacrylate	16 g	
	tetraethylene glycol diacrylate	13 g	
	N-methyldiethanolamine	0.05 g	
45	Victoria Blue	0.04 g	45
	2,4-dimethylthioxanthone	0.5 g	
	4-methylbenzophenone oxime acetate	1.5 g	
	o-toluenesulfonamide	2.4 g	
	p-toluenesulfonamide	3.6 g	
50	benzotriazole	0.2 g	50
	methylethyl ketone	120 g	

A high-quality image was obtained until the 7th step in the same manner as in Example 3.

Example 9

A photopolymerizable resin composition was obtained by mixing the following components:

	poly(methyl methacrylate)	50 g	
5	trimethylolpropane triacrylate	16 g	5
	tetraethylene glycol diacrylate	13 g	
	N-methyldiethanolamine	0.05 g	
	Victoria Blue	0.04 g	
	2,4-diethylthioxanthone	0.5 g	
10	4-methylbenzophenone oxime acetate	1.5 g	10
	o-toluenesulfonamide	2.4 g	
	p-toluenesulfonamide	3.6 g	
	benzotriazole	0.2 g	
	methyl ethyl ketone	120 g	
15			15
	A high-quality image was obtained until the 8th step in the same manner as in Example 3.		

Comparative Example 1

The following components were mixed together:

20	poly(methyl methacrylate)	50 g	20
	trimethylolpropane triacrylate	15 g	
	tetraethylene glycol diacrylate	10 g	
	benzotriazole	0.2 g	
25	N-methyldiethanolamine	0.05 g	25
	Victoria Blue	0.04 g	
	2-chlorothioxanthone	0.1 g	
	methyl ethyl ketone	120 g	
30	After the application, drying and exposure conducted in the same manner as in Example 3, no image could be obtained at all.		

Comparative Example 2

The following components were mixed together:

35	poly(methyl methacrylate)	50 g	35
	trimethylolpropane triacrylate	15 g	
	tetraethylene glycol diacrylate	10 g	
	benzotriazole	0.2 g	
40	N-methyldiethanolamine	0.05 g	40
	Victoria Blue	0.04 g	
	4-methylbenzophenone oxime acetate	1.5 g	
	methyl ethyl ketone	120 g	
45	After the application, drying and exposure to a light of an ultra-high pressure mercury lamp conducted in the same manner as in Example 3, no image could be obtained at all.		

CLAIMS

1. A composition for photopolymerization initiation which comprises:
(A) thioxanthone or a derivative thereof of the general formula



- 15 in which Ar₁ and Ar₂ each are an aromatic ring or a substituted aromatic ring,
and
(B) an oxime ester of the general formula:



- 25 wherein R₁ and R₂ may be the same or different and each represents an alkyl group having 1 to 10 carbon atoms, a substituted or unsubstituted phenyl, naphthyl, anthryl, pyridyl or quinolyl group or R₁ and R₂ may be bonded together to form a ring, and R₃ represents an alkyl group having 1 to 5 carbon atoms, or a substituted or unsubstituted aryl group.

- 30 2. An initiator composition as claimed in claim 1, which comprises the compounds (A) and (B) in a weight ratio of 1:100 to 10:1.

3. A composition as claimed in either of claims 1 or 2, wherein the groups R₁ and R₂ of the oxime ester (B) are the same or different and each is an alkyl group having 1 to 3 carbon atoms, a phenyl, naphthyl, anthryl, 2-pyridyl, 2-quinolyl, cyclohexanone, anthrone, 10-methyl-9(10H)-acridone, piperidone, or 9-fluorenone group unsubstituted or substituted with an alkyl group having 1 to 10 carbon atoms, an alkoxy group having 1 to 5 carbon atoms, a halogen, an amino, a dimethylamino, a diethylamino or a mercapto group, and R₃ is any of methyl, ethyl, propyl, i-propyl, butyl, t-butyl, phenyl, benzoyl, toluoyl, chlorobenzoyl, nitrobenzoyl, methoxybenzoyl, naphthyl or anthroquinone.

4. A composition as claimed in claim 3, in which the phenyl, naphthyl, anthryl, 2-pyridyl, 2-quinolyl, cyclohexanone, anthrone, 10-methyl-9(10H)-acridone, piperidone, or 9-fluorenone group may be substituted with an alkyl group having 1 to 5 carbon atoms.

5. A composition as claimed in any of claims 1 to 4, in which component A comprises at least one of 2-chlorothioxanthone, 2,4-diethylthioxanthone, 2,4-dimethylthioxane and thioxane.

6. A composition as claimed in any of claims 1 to 5, in which component B comprises 4-methylbenzophenone.

7. A photopolymerizable resin composition which comprises 5 to 80 percent by weight of one or more photopolymerizable compounds having a double bond for polymerization, 0.01 to 20 percent by weight of a composition as claimed in any of claims 1 to 6, 0 to 94.99 percent of a polymer binder, and the balance, if any, comprising other photopolymerizable resin composition additives.

8. A photopolymerizable resin composition as claimed in claim 7, comprising from 1 to 10 wt. % of the composition defined in any of claims 1 to 6.

9. A photopolymerizable resin composition as claimed in claims 7 or 8, comprising from 10 to 50 wt. % of the at least one photopolymerizable compound.

10. A photopolymerizable resin composition as claimed in any of claims 7 to 9 which comprises 30 to 100 weight percent in total of the photopolymerizable compound and the initiator composition as claimed in any of claims 1 to 6, and zero to 70 weight percent of the polymer binder.

11. A photopolymerizable resin composition as claimed in any of claims 7 to 10, wherein the polymer binder comprises polymethyl methacrylate.

12. A photopolymerizable resin composition as claimed in any of claims 7 to 11, wherein any additive used comprises Victoria Blue.

13. A photopolymerizable resin composition substantially as hereinbefore described with reference to the accompanying examples.

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